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(54) PRODUCTION OF MICROCELLULAR POLYESTER FILM

(57) Abstract:

PROBLEM TO BE SOLVED: To provide a polyester film wherein the edge scraps can be effectively reclaimed, the amount of scraps which are disposed of can be markedly reduced, and the state of existence of microcells is excellent.

SOLUTION: The production process consists of preparing a film obtained by stretching a sheet prepared by coextruding a polyester A as a core and a polyester B as each end in at least the transverse direction and cutting off the ends derived from polyester B from the film. The following relationships I to III should be simultaneously satisfied: formula I: $5 \leq An(\text{wt.\%}) \leq 45$, formula II: $0 \leq Bn(\text{wt.\%}) \leq 10$, and formula III: $An(\text{wt.\%}) > Bn(\text{wt.\%}) + 5$ (wherein An is the content of an incompatible thermoplastic resin in the polyester A, and Bn is the content of an incompatible thermoplastic resin in the polyester B).

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CLAIMS

[Claim(s)]

[Claim 1] In the manufacture approach of the polyester film which carries out cutting clearance of the film both ends which originate in said polyester B from the film which extends at least the sheet by which used Polyester A as the center section and the co-extrusion was carried out considering Polyester B as both ends in a longitudinal direction, and is obtained Content An of the immiscible nature thermoplastics in said polyester A Content Bn of the immiscible nature thermoplastics in said polyester B The manufacture approach of the detailed air-bubbles content polyester film characterized by satisfying following type ** - ** simultaneously.

[Equation 1] $5 \leq An(\% \text{ of the weight}) \leq 45$ —**
 $0 \leq Bn(\% \text{ of the weight}) \leq 10$ —**
 $An(\% \text{ of the weight}) > Bn(\% \text{ of the weight}) + 5$ — ** — [Claim 2] Intrinsic viscosity IVA of the film part originating in Polyester A Intrinsic viscosity IVB of 0.45 to 0.70, and the film ends part originating in Polyester B It is 0.60–0.85 and is IVB–IVA. The manufacture approach of the detailed air-bubbles content polyester film according to claim 1 characterized by being 0–0.15.

[Claim 3] The manufacture approach of the detailed air-bubbles content polyester film according to claim 1 or 2 characterized by immiscible nature thermoplastics being at least one sort chosen from polypropylene, the poly methyl pentene, and polystyrene.

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DETAILED DESCRIPTION**[Detailed Description of the Invention]****[0001]**

[Field of the Invention] This invention relates to the manufacture approach of detailed air-bubbles content polyester film.

[0002]

[Description of the Prior Art] Since it has various kinds of properties with sufficient balance highly and excels in respect of cost performance, polyester film is widely used as industrial materials. And the detailed air-bubbles content polyester film which film-sized the polyester which blended immiscible thermoplastics is lightweight, and is used for the application of synthetic papers, such as for example, pasteboard for a seal print, a magnetic card, etc. as a white opaque film equipped with cushioning properties. The cushioning properties are respected and especially detailed air-bubbles content polyester film is used as the television paper and pasteboard (release paper) of the product which printed the image and was used as the seal. If the frame and background of hope are chosen and a pose is made as the example of an activity, the so-called activity in the amusement machine which can take a photograph and process on that spot, and can create a photograph-of-his-face seal will be mentioned.

[0003] By the way, generally in manufacture of detailed air-bubbles content polyester film, the film is grasped with a tenter clip in the case of a longitudinal direction drawing. Generally the both ends of the film volume riser containing this tenter clip grasping section are called a handle part. The slit of this handle part film is carried out, and playback or abolition processing is performed as a scrap. Although it is desirable from the field of raw material cost to carry out reuse of the handle part film from consideration of an environment, in using a handle part film for manufacture of detailed air-bubbles content polyester film as a playback raw material, there are the following problems. That is, to the polyester which existed in the scrap film, the resin of immiscible nature deteriorated by the heat history in a playback process, and has lost the generation ability of enough detailed air bubbles. It is in the inclination checked to generation of the detailed air bubbles by the immiscible nature resin newly added to the surprising thing about it and by this playback raw material combination system. For this reason, in order not to reduce generation of detailed air bubbles, combination of a playback raw material is limited very a little, and most handle part films must be discarded.

[0004]

[Problem(s) to be Solved by the Invention] The efficient reuse of a handle part scrap of this invention becomes possible, it can reduce remarkably the amount of the scrap discarded simultaneously, and makes it a technical problem to offer the polyester film which was moreover excellent in the condition of content of detailed air bubbles.

[0005]

[Means for Solving the Problem] That the above-mentioned technical problem should be solved, wholeheartedly, as a result of examination, this invention person finds out that the above-mentioned technical problem can be solved easily, and came to complete this invention by adopting the manufacture approach which serves as a specific configuration. Namely, the summary of this invention uses Polyester A as a center section. In the manufacture approach of

the polyester film which carries out cutting clearance of the film both ends which originate in said polyester B from the film which extends at least the sheet by which the co-extrusion was carried out considering Polyester B as both ends in a longitudinal direction, and is obtained Content An of the immiscible nature thermoplastics in said polyester A The content Bn of the immiscible nature thermoplastics in said polyester B consists in the manufacture approach of the detailed air-bubbles content polyester film characterized by satisfying following type ** - ** simultaneously.

[0006]

[Equation 2] $5 \leq An(\% \text{ of the weight}) \leq 45$ — ** $0 \leq Bn(\% \text{ of the weight}) \leq 10$ — ** $An(\% \text{ of the weight}) > Bn(\% \text{ of the weight}) + 5$ — ** [0007]

[Embodiment of the Invention] Hereafter, this invention is explained to a detail. In this invention, the polyester which constitutes each part of the sheet by which the co-extrusion was carried out is polyester obtained considering aromatic series dicarboxylic acid, or its ester and glycol as a main start raw material, and 80% or more of a repeat structural unit points out an ethylene terephthalate unit or ethylene -2, and the polyester with which it has 6-naphthalate unit. And as long as it is under the condition which does not deviate from the above-mentioned range, other third components may be contained.

[0008] As an aromatic series dicarboxylic acid component which constitutes polyester, kinds, such as isophthalic acid, a phthalic acid, an adipic acid, a sebacic acid, and hydroxy acid (for example, p-oxyethoxy benzoic acid etc.), or two sorts or more can be used in addition to terephthalic-acid and 2, and 6-naphthalene dicarboxylic acid. As a glycol component, kinds, such as a diethylene glycol, propylene glycol, butanediol, 1, 4-cyclohexane dimethanol, and neopentyl glycol, or two sorts or more can be used in addition to ethylene glycol.

[0009] By the manufacture approach of this invention, it faces carrying out melting extrusion to the shape of a sheet, and sees crosswise [of a sheet], and each polyester melting object is made to join in its this side in for example, T dice, and is extruded so that it may become polyester B / polyester A / polyester B. In this case, it is more desirable to consider as the same raw material, since it becomes unnecessary to take crosswise object sex difference into consideration in case it is easier to divide passage from what was fused with one extruder, and to supply both ends and it extends at an after process, although the polyester B set by the ends of Polyester A does not necessarily need to be based on the same raw material.

[0010] The detailed air-bubbles content polyester film by this invention is obtained by extending the polyester which contains the thermoplastics (it may be hereafter written as immiscible nature resin) which is immiscible nature to polyester to at least 1 shaft orientations. The content An of the immiscible nature resin of the polyester A section is a rate to the total quantity with polyester, and let it preferably be 10 – 25% of the weight of the range still more preferably five to 35% of the weight five to 45% of the weight. An Since there are too few amounts of the air bubbles formed into a film when it is less than 5 % of the weight, the film which was fully lightweight-ized and was equipped with cushioning properties cannot be manufactured. On the other hand, it is An. When exceeding 45 % of the weight, the mechanical strength and thermal stability of a film run short, and, moreover, the relative roughness on the front face of a film becomes large too much. This film becomes unsuitable at the application of the television paper in which aesthetic property and clear printing nature are demanded. Moreover, An When exceeding 45 % of the weight, the problem on the productivity that film fracture occurs frequently at the time of a drawing may arise.

[0011] the consistency of the film part originating in BORIESUTERU A — usually — 0.40 – 1.30 g/cm³ — desirable — 0.60 – 1.20 g/cm³ — further — desirable — 0.70 – 1.10 g/cm³ It becomes the range. Moreover, in the manufacture approach of this invention, the handle part film of a constant rate is surely generated as a scrap, and playback or abolition processing is performed. That is, the both ends of the film volume riser containing the tenter grasping section in a horizontal drawing hit this. Here, as for consideration of the environment where the advantage in respect of raw material cost and the amount of abolition of a scrap are reduced to a handle part film, it is desirable that reuse is carried out.

[0012] It is [as opposed to / in order to make possible reuse of the part which originates in the

polyester B used as the ends (lug) section of a product volume riser in this invention / Polyester B] the content Bn of immiscible thermoplastics. It may be 0 – 1 % of the weight still more preferably zero to 3% of the weight preferably zero to 10% of the weight. Bn When exceeding 10 % of the weight, in case the B section film is regenerated, the problem from which degradation of the immiscible nature resin by the heat history becomes remarkable arises. If this playback raw material that deteriorated is blended with the B section, at the time of production, film fracture occurs frequently and a product cannot be extracted. Moreover, since sufficient generation of detailed air bubbles is checked by the effect of the immiscible nature resin which deteriorated in case it blends with the polyester A section, the loadings will be limited very a little.

[0013] The consistency of the film part originating in Polyester B is usually 3 1.10g/cm. It is 1.35 – 1.50 g/cm³ preferably [it is desirable and] to 1.25 – 1.50 g/cm³ and a pan above. It is the range. It sets to this invention and is An. Bn It is required for satisfying the above-mentioned amount relation and coincidence to fill the relation of An >Bn+5. When not filling this relation, the effectiveness of the reuse acceleration by reducing the amount of immiscible nature resin of the polyester B section becomes indefinite.

[0014] Although polystyrene besides polyolefines, such as polyethylene, polypropylene, the poly methyl pentene, and the poly methylbutene, a polycarbonate, polyphenylene sulfide, liquid crystal polyester, etc. are mentioned to the polyester which can be used by this invention as an example of immiscible thermoplastics, in these, polypropylene, the poly methyl pentene, and polystyrene are polypropylene desirable still more preferably from a viewpoint of cost or productivity. In addition, in the following explanation, to polyester, it is polypropylene, with immiscible thermoplastics is represented.

[0015] As the above-mentioned polypropylene, more than 95 mol % and the crystalline polypropylene homopolymer in which more than 98 mol % has a propylene unit preferably are usually desirable. In a film production process, polypropylene carries out bleed out of the case of amorphous polypropylene to the front face of a non-orientation polyester sheet, and front faces, such as a cooling drum and a drawing roll, are easy to be polluted. Moreover, in the case of the polypropylene with which copolymerization for example, of the ethylene units other than a propylene unit is carried out exceeding five-mol %, generation of detailed air bubbles tends to run short.

[0016] the melt flow index (MFI) of the above-mentioned polypropylene — usually — 0.5–30g/— it is preferably chosen from the range for 1.0–15g / 10 minutes for 10 minutes. When there is an inclination for fracture to become easy to take place at the time of a drawing by the air bubbles generated when MFI(s) are 0.5g / less than 10 minutes becoming large too much and MFI exceeds 30g / 10 minutes, the homogeneity of a film consistency with time tends to be inferior, and productivity may get worse with a production line.

[0017] Next, intrinsic viscosity IVA of the film part originating in Polyester A It is desirable that it is 0.45–0.70. When intrinsic viscosity is less than 0.45, there is a possibility that there may be an inclination for film fracture to become easy to take place at the time of film production, and for the magnitude of air bubbles to become an ununiformity, and for control of a consistency to become difficult, and productivity may fall. On the other hand, when intrinsic viscosity exceeds 0.70, there is an inclination for generation of the detailed air bubbles in a film to decrease.

[0018] Intrinsic viscosity IVB of the film part originating in Polyester B It is desirable that it is 0.60–0.85. When intrinsic viscosity is less than 0.60, there is an inclination for film fracture to become easy to take place, at the time of film production. On the other hand, when intrinsic viscosity exceeds 0.85, the productivity at the time of polyester raw material manufacture may fall. further — the difference (IVB–IVA) of the intrinsic viscosity of the film parts of Polyester A and B origin — 0–0.15 — a certain thing is desirable. When this difference is minus, there is an inclination film fracture becomes easy to produce from near the clip grasping section. On the other hand, when this difference exceeds 0.15, there is an inclination the film fracture from an A/B interface becomes easy to produce.

[0019] In the manufacturing method of this invention, the relative roughness of the consistency of a detailed air-bubbles content film, cushioning properties, and a front face can be adjusted by controlling the magnitude of the distributed particle size of the polypropylene formed at a melting

extrusion process. In order to consider as the range of a request of the distributed particle size, it is desirable to make a surfactant contain in polyester. As the above-mentioned surface active agent, although an anion system surface active agent, a cation system surface active agent, an amphoteric surface active agent, a nonionic surfactant, etc. are mentioned, in these, a nonionic surfactant especially a silicone system surface active agent, and a polyalkylene glycol are desirable. As a silicone system surfactant, an organopolysiloxane-polyoxyalkylene copolymer, the alkenyl siloxane which has a polyoxyalkylene side chain are mentioned. As a polyalkylene glycol, the copolymer of a polyethylene glycol, a polypropylene glycol, ethylene glycol, and propylene glycol etc. is mentioned. Usually let preferably the content of the surfactant in the total raw material for constituting an A horizon be 0.01 – 1.0% of the weight of the range 0.005 to 2.0% of the weight. When the content of a surfactant exceeds 2.0 % of the weight, in addition to the effectiveness of a surfactant not improving any longer, the trouble in an extruder and degradation of polyester may be caused. On the other hand, the content of a surfactant may be insufficient for the manifestation of the effectiveness at less than 0.005 % of the weight.

[0020] On the film obtained according to the manufacturing method of this invention, in order to give white nature and concealment nature, a titanium dioxide, a barium sulfate, and other well-known white pigments may be added. In order to raise a whiteness degree further furthermore, it is also effective to add a fluorescent brightener. Moreover, it is desirable from a viewpoint of raw material cost to perform addition of white pigments and a fluorescent brightener only in the polyester A section.

[0021] Although two or more kinds of white pigments can also be made to contain, it is desirable to make a titanium dioxide or a barium sulfate contain at least in that case. The desirable content of the white pigments in the polyester A section is 0.5 – 20 % of the weight, and a still more desirable content is 1.0 – 15 % of the weight. As an example of the fluorescent brightener which can be used suitably, the Ciba-Geigy goods "YUBITEKKU", the Eastman goods "alumnus-1", etc. are mentioned. The desirable content of the fluorescent brightener in the polyester A section is 0 – 0.30% of the weight of the range.

[0022] In addition, in this invention, additives other than the aforementioned white pigments and a fluorescent brightener, such as a well-known antioxidant, a thermostabilizer, lubricant, an antistatic agent, a color, and a pigment, can be blended if needed into polyester or polypropylene. The approach of doubling and extruding the melting object which consists of the polyester A section and the polyester B section at least so that it may see crosswise [of a sheet] as mentioned above and the B section may serve as ends can apply the approach of put together currently indicated by the approach learned conventionally, for example, JP,55-118832,A, JP,1-118428,A, JP,8-207119,A, etc.

[0023] After it makes polymer melt join in advance and it carries out a co-extrusion by these approaches, it extends by the tenter method in a longitudinal direction at least. However, while forming detailed air bubbles good, in order to satisfy the reinforcement and dimensional stability of a film moderately, the biaxial-stretching approach and the heat treatment approach are usually used together. Hereafter, an example at the time of using biaxial stretching for the manufacture approach of this invention is explained to a detail. First, the raw material of the combination to each part of the cross direction is supplied to the extruder corresponding to each section of the equipment for launching for co-extrusion. That is, after usually throwing raw material resin into three sets of 2 or extruders and carrying out melting kneading for every extruder line, it leads to the multi-manifold or feed block which draws the melt flow of the three sections, and extrudes as a melting sheet from a die.

[0024] In order to see crosswise [of a sheet] and to specifically make the configuration of each part into B/A/B, each raw material which constitutes the A section and the B section is extruded from one extruder, respectively, and, generally the method of dividing a B horizon melt line on the way is used. Quantum feeders, such as a gear pump, are installed in the melt line after division, the flow rate of the polymer melt of the B section doubled with the ends of the A section is adjusted to each, and the width of face of the B section is controlled. Of course, it is also effective on width-of-face adjustment of the A section, and thickness accommodation to install a quantum feeder in the melt line of A section polyester. Next, quenching solidification of

the melting sheet extruded from the die is carried out so that it may become the temperature below glass transition temperature by revolution cooling drum lifting, and the non-orientation sheet of amorphous state is obtained substantially. In this case, in order to raise the smoothness of a sheet, and the cooling effect, it is desirable to raise the adhesion of a sheet and a revolution cooling drum, and electrostatic impression contact printing and/or liquid spreading contact printing are preferably adopted in this invention.

[0025] Subsequently, the obtained sheet is extended and film-sized to 2 shaft orientations. This drawing is made to generate the detailed air bubbles of the film part of the polyester A origin in the manufacture approach of this invention. First, said non-extended sheet is 70-150 degrees C usually preferably extended to an one direction (lengthwise direction) under the condition of draw magnification of being 3.0 to 5 times many as this the drawing temperature of 75-130 degrees C, and usually [2.5 to 6 times as many as this]. The drawing machine of a roll and a tenter method can be used for this drawing. Subsequently, under the condition of draw magnification of being 3.0 to 5 times many as this, it extends in the drawing temperature of 80-140 degrees C, and the direction (longitudinal direction) which intersects perpendicularly with the first step, and 75-150 degrees C of biaxial oriented films are usually obtained 2.5 to 6 times preferably. The drawing machine of a tenter method can be used for this drawing.

[0026] Although the approach of extending the above-mentioned one direction in two or more steps is also employable, it is desirable to go into the range which final draw magnification described above also in that case. Moreover, it is also possible to carry out simultaneous biaxial stretching of said non-extended sheet so that an area scale factor may increase 7 to 30 times. Heat treatment is 150-250 degrees C, and is performed for [1 second -] 5 minutes under less than 30% of expanding, limit contraction, or fixed-length. After biaxial stretching, after performing a re-drawing to a lengthwise direction 1.05 to 2.0 times at the temperature of 110 more degrees C - 180 degrees C, the approach of heat-treating can also be taken. Under the present circumstances, it is also possible to adopt suitably technique, such as a back minute scale-factor length drawing before front [a re-length drawing] heat setting, after [a re-length drawing] length relaxation, and a re-length drawing. Moreover, a re-drawing may be similarly performed in a longitudinal direction. Moreover, various kinds of surface treatment etc. may be performed within a film production process if needed.

[0027] The detailed air-bubbles content polyester film by the manufacture approach of this invention is not necessarily a single layer. When the polyester A section consists of two or more layers, what is necessary is just the detailed air-bubbles content layer which contains the thermoplastics of immiscible nature further at least. Moreover, 20-250 micrometers of thickness of detailed air-bubbles content polyester film usually serve as the range of 50-200 micrometers preferably. Moreover, taking advantage of the description, it is used suitable for printer television paper, the pasteboard for a seal print, a label, the recording paper, a poster, a lithography plate, wrapping, a tag, etc.

[0028]

[Example] Hereafter, although an example explains this invention to a detail further, this invention is not limited to the following examples, unless the summary is exceeded. In addition, the assessment approach in an example and the example of a comparison is as being shown below. Moreover, all the "sections" of the component ratio in an example and the example of a comparison means the "weight section", and a silicone system surfactant shows the product "SH193" made from Toray Industries Silicone.

(1) Intrinsic viscosity of polyester [η] (dl/g)

The mixed solvent of a phenol / tetrachloroethane:50 / 50 (weight ratio) was added and dissolved in polyester by the 100ml ratio to polyester 1g which removed other immiscible polymer components and white pigments, and it measured at 30 degrees C.

(2) Melt flow index M-F-I (g/10min)

JIS It measured according to K-6758-1981. It is shown that the melt viscosity of a polymer is so low that this value is high.

[0029] After M-F-I blended 13 % of the weight of crystalline polypropylene chips, 2.4 % of the weight of titanium oxide, and 0.1 % of the weight of silicone system surface active agents for

10g / 10 minutes to the polyethylene terephthalate chip of example 1 intrinsic viscosity 0.69, it blended to homogeneity and the polyester raw material X was adjusted. The polyethylene terephthalate chip of intrinsic viscosity 0.66 was adjusted separately, and it considered as the raw material Y.

[0030] Each above-mentioned raw material was respectively fused at 280 degrees C after supplying in a separate extruder, each acquired melting object was led to T dice, and it was made to join in advance so that it may become the configuration of raw material Y / raw material X / raw material Y crosswise. In addition, the die-length ratio of the cross direction of the sheet of each part was set to 1:8:1. It extruded in the shape of a slit by longitudinal direction co-extrusion, and cooled on the 30-degree C cast roll, and the non-orientation sheet was obtained. And after carrying out a roll drawing 3.4 times at 85 degrees C, further, the tenter drawing was carried out 3.1 times at 110 degrees C, heat treatment was further performed for 5 seconds at 230 degrees C within the tenter drawing machine to the flow direction (lengthwise direction) of a film at the longitudinal direction, and the biaxial oriented film containing detailed air bubbles was obtained in it into the part which originates in a raw material X eventually. The object which carried out the slit of the part originating in the raw material Y of the obtained film, and regenerated it was what can be again used suitably as an ingredient of a raw material Y.

[0031] It faced fusing each raw material with a separate extruder, and extruding from T dice, and was made to join in advance in example 2 example 1, so that it may become the configuration of raw material Y/(field where the laminating of a raw material X and the raw material Y was carried out to the order of YXY in the thickness direction) / raw material Y crosswise. In addition, the crosswise die-length ratio of the sheet of each part is 1:8:1, and it was made, as for the field where the laminating of the center section is carried out, for the flow rate of the melting object of a raw material X and a raw material Y to be set to 14:1. The biaxial orientation laminated film which contains detailed air bubbles like an example 1 henceforth was obtained. The object which carried out the slit of the part originating in the raw material Y of the obtained film, and regenerated it was what can be again used suitably as an ingredient of a raw material Y.

[0032] The raw material X of example of comparison 1 example 1 was fused, it extruded from T dice with the single configuration crosswise, and the biaxial oriented film which contains detailed air bubbles like an example 1 henceforth was obtained. When the scrap which carried out the slit of the handle part of the obtained film was regenerated and the whole quantity was blended into the raw material X, it resulted in reducing generation of the detailed air bubbles after film-izing. In order to maintain the amount of generation of detailed air bubbles, the reuse of a scrap was a bad film of a raw material yield in which parenchyma is impossible.

[0033] In example 3 example 1, the biaxial oriented film which contains detailed air bubbles similarly was obtained except having set to 0.74 intrinsic viscosity of the polyethylene terephthalate chip used for a raw material X, and having set to 0.64 intrinsic viscosity of the polyethylene terephthalate chip used for a raw material Y. It was the film which film fracture is sporadic from near the tenter clip grasping section, and is a little inferior in a processing yield as compared with an example 1 at the time of production.

[0034] In example 4 example 1, the biaxial oriented film which contains detailed air bubbles similarly was obtained except having set to 0.74 intrinsic viscosity of the polyethylene terephthalate chip used for a raw material X, and having set to 0.84 intrinsic viscosity of the polyethylene terephthalate chip used for a raw material Y. It was the film which film fracture is sporadic from near the interface of a raw material X and a raw material Y, and is a little inferior in a processing yield as compared with an example 1 at the time of production. The result obtained above is collectively shown in the following table 1.

[0035]

[A table 1]

表1

		ポリエステルの固有粘度		生産時の フィルム 破断
		原 料	フィルム	
実施例1	中央部	0. 69	0. 59	な し
	両端部	0. 66	0. 63	
実施例2	中央部	0. 69	0. 59	な し
	両端部	0. 66	0. 63	
比較例1		0. 69	0. 59	な し
実施例3	中央部	0. 74	0. 63	散 発
	両端部	0. 64	0. 61	
実施例4	中央部	0. 74	0. 63	散 発
	両端部	0. 84	0. 80	

[0036]

[Effect of the Invention] Deteriorating in response to the heat history while the thermoplastics of the immiscible nature in a scrap regenerating could not but discharge the handle part scrap conventionally generated in the production process of detailed air-bubbles content polyester film by abolition processing owing to. However, it obtained reducing remarkably the amount of the scrap which the efficient reuse of a handle part scrap of becomes possible, and is simultaneously discarded by applying the manufacture approach of this invention. When the merit in respect of a production cost and the good effect on an environment are taken into consideration, the industrial value is dramatically high.

[Translation done.]